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Processing and Properties of Mechanical Alloyed $Al_{93}Fe_3Cr_2Ti_2$ Alloys

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ABSTRACT

Nanostructured $Al_{93}Fe_3Ti_2Cr_2$ alloys were prepared via mechanical alloying (MA) starting from elemental powders, followed by extrusion to form bulk material. The microstructure and phase formation as a function of the milling time and the concentration of the process-control agent were investigated. The thermal stability of the mechanical alloyed powder was also studied. The bulk material extruded from the mechanical alloyed powder exhibited superior strength with good ductility at both ambient and elevated temperatures under compressive loading conditions. The enhanced strength at both ambient and elevated temperatures was attributed to the retention of ultrafine grains (fcc-Al < 100 nm), the formation of supersaturated solid solutions, and the presence of nanoscale intermetallic precipitates.

INTRODUCTION

Recent progress in studies of bulk amorphous, quasicrystalline and nanocrystalline alloys has resulted in substantial improvements in the strength of Al-based alloys [1-10]. Bulk Al alloys with tensile strengths as high as 800 – 1000 MPa and reasonable ductility (2 to 10% tensile elongation) have been demonstrated recently [8-10]. Many of these Al alloys derive their superior strengths from either a microstructure of nanoscale fcc-Al crystals distributed in an amorphous matrix [7,9] or a microstructure containing a mixture of nanoscale fcc-Al particles and intermetallic aluminides [10].

It is noted that nearly all of the reported amorphous, quasicrystalline and nanocrystalline Al alloys that possess high strengths coupled with good ductility are produced through rapid quenching approaches such as melt spinning or gas atomization, followed by extrusion [1-13]. One exception to this is the recent work by Hayes, et al. [14] who have formed nanocrystalline Al-10Ti-2Cu alloys via extrusion and ball milling of gas-atomized Al-Ti-Cu alloy powder within a liquid nitrogen medium.

In this study we have investigated the feasibility of making nanocrystalline $Al_{93}Fe_3Ti_2Cr_2$ alloys via mechanical alloying (MA) starting from elemental powders, and examined the potential of the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy for high-temperature structural applications. $Al_{93}Fe_3Ti_2Cr_2$ alloys have been chosen for investigation because Fe, Cr and Ti all have very low diffusivity and equilibrium solubility in fcc-Al (0.03 at.% Fe, > 0.01 at.% Cr, and > 0.2 at.% Ti) [15,16]. Low diffusivity and solubility are the basic requirements for preventing Ostwald ripening [17]. Thus, $Al_{93}Fe_3Ti_2Cr_2$ alloys are expected to have good microstructural stability at elevated temperatures and thus superior high-temperature strengths. $Al_{93}Fe_3Ti_2Cr_2$ alloy has been prepared previously by Inoue, et al. [5] using the gas atomization approach followed by extrusion. They have indeed

found that this alloy possesses superior high temperature strength. The reported ultimate tensile strength at 300^0C is 360 MPa [5]. This high temperature strength compares very favorably against the current commercial Al alloys, most of which lose their useful strengths at temperatures above 250^0C (typically becoming lower than 200 MPa) [18].

The potential advantage using MA over rapid quenching (i.e., gas-atomization) to prepare nanocrystalline $Al_{93}Fe_3Ti_2Cr_2$ alloys is the possibility of making nanocrystalline Al matrix composites with nano-reinforcements (i.e., nano/nano-Al composites) through blending the elemental constituents of the metallic matrix or pre-alloyed metallic powder with insoluble nano-reinforcements such as nano-SiC particles. Nano/nano-Al composites are expected to have better microstructural stability and thus higher elevated temperature strengths than those exhibited by the corresponding nanocrystalline Al alloys.

EXPERIMENTAL PROCEDURE

Crystalline elemental powders were used to prepare Al alloys with a nominal composition of $Al_{93}Fe_3Ti_2Cr_2$. The aluminum powder had a purity of 99.5 wt% with a mean particle size of 70 μm, while the corresponding values for iron, chromium and titanium powders used were 99.0%, 98.5% and 99.5% as well as 50, 30 and 30 μm, respectively. Mechanical alloying was performed in a Szegvari attritor. The canister of the attritor was made of a stainless steel and the charge consisted of stainless steel balls with a diameter of 4.76 mm. A ball-to-powder weight ratio of 20:1 and a milling speed of 600 RPM were employed in all the experiments. During milling the canister was cooled using circulation water with a flow rate of about 770 ml/min throughout the process and an argon atmosphere was employed in all the experiments. To prevent excessive cold welding of Al alloys, stearic acid [$CH_3(CH_2)_{16}COOH$] was added to the powder mixture as the process control agent (PCA).

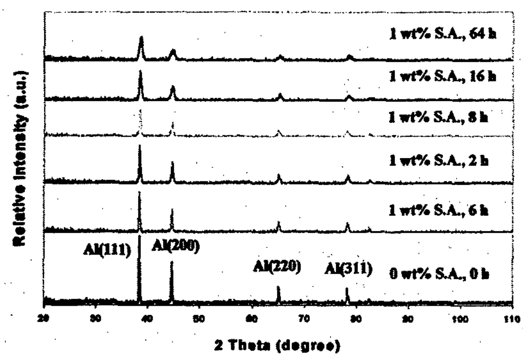
The microstructural evolution, formation of supersaturated solid solutions and grain size reduction in the powder particles as a function of the milling time and the weight percent of the process control agent were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The detailed procedure for estimating the crystallite size and internal strain using XRD can be found elsewhere [19,20]. Based on these characterizations, the mechanical alloyed powder milled for 30 hours with the addition of 1.2 wt% of stearic acid was selected for investigation of grain growth and phase transformation when exposed to elevated temperatures. The same mechanical alloyed powder was also extruded to form bulk materials, and compressive specimens were made from these extruded materials [21]. Both ends of the compressive specimens were coated with MoS_2 before tests and the strain rate employed was 10^{-3} sec^{-1} for all the tests. All the compressive tests were displacement controlled and terminated if the specimen did not fracture at -0.45 true strain. The test temperature investigated ranged from 25 to 400^0C.

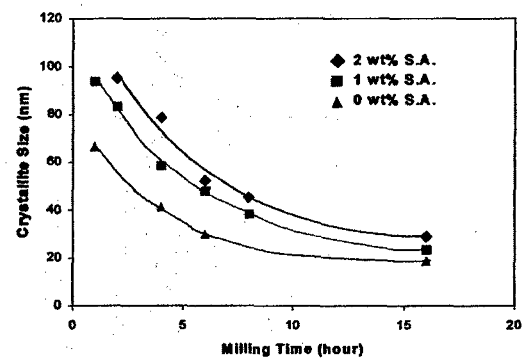
RESULTS AND DISCUSSION

Fig. 1 shows XRD patterns of the Al alloy powder with 1 wt% stearic acid as a function of milling time. Note that Fe, Cr and Ti reflections are hardly discernable even for the powder mixture without milling because Fe, Cr and Ti concentrations are low and some of their peaks overlap with Al peaks. Thus, the detection of the formation of Al-Fe-Cr-Ti alloys and other structural information could only rely on the measurement of changes in Al reflections. A close examination of Fig. 1 reveals the following two features:

1) The peak position of Al reflections shifts to higher 2θ angles as the milling time increases. This suggests the formation of Al-based solid solutions and an increase in the solute concentration as the milling time increases.
2) All aluminum reflections exhibit broadening even after only 2-hours of milling, indicating the grain size reduction and possibly the introduction of internal strains.

Based on the peak broadening of XRD reflections, the crystallite size of fcc-Al in the Al powder mixture as a function of milling time have been estimated. The results are shown in Fig. 2, which indicates that the calculated crystallite size decreases rapidly at the early stage of milling and levels off at prolonged milling time. Furthermore, the presence of PCA decreases the grain refinement rate. The more PCA in the powder, the smaller the decrease in the crystallite size. In spite of this phenomenon, after 16 hours of milling the crystallite size of fcc-Al in all the aluminum powder mixtures with and without PCA has been reduced to a range of 20 to 30 nanometers.





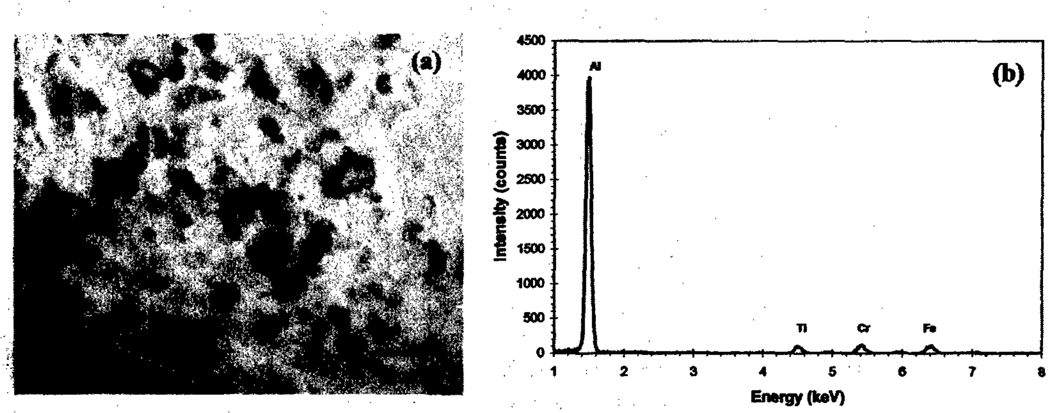


Fig. 3 (a) A TEM bright-field image and (b) a TEM EDS spectrum from one fcc-Al grain of the Al alloy powder milled for 30 hours.

The formation of nano-grains and Al-based solid solutions from mechanical alloying are confirmed by the TEM analysis. As shown in Fig. 3(a), the TEM bright-field image reveals that the Al alloy powder particles milled with 1.2 wt% stearic acid for 30 hours are composed of nano-grains with sizes ranging from 6 to 45 nm. The analysis using the energy dispersive spectrometry

(EDS) attached to the TEM with an electron beam of 3.5 nm verifies that nano-grained fcc-Al is indeed the Al-based solid solution containing Fe, Cr and Ti alloying elements [Fig. 3(b)]. These mechanical alloyed powder particles exhibit phase transformation when exposed to elevated temperatures. Shown in Fig. 4 are XRD patterns of the mechanical alloyed powder as a function of annealing temperature. It is noted that the as-milled, nanocrystalline, supersaturated fcc-Al solid solution is stable up to 300^0C above which precipitates, Al_6Fe, Al_3Ti, $Al_{13}Fe_4$ and $Al_{13}Cr_2$, have been observed. Details of the phase transformation sequence can be found in Reference 20.

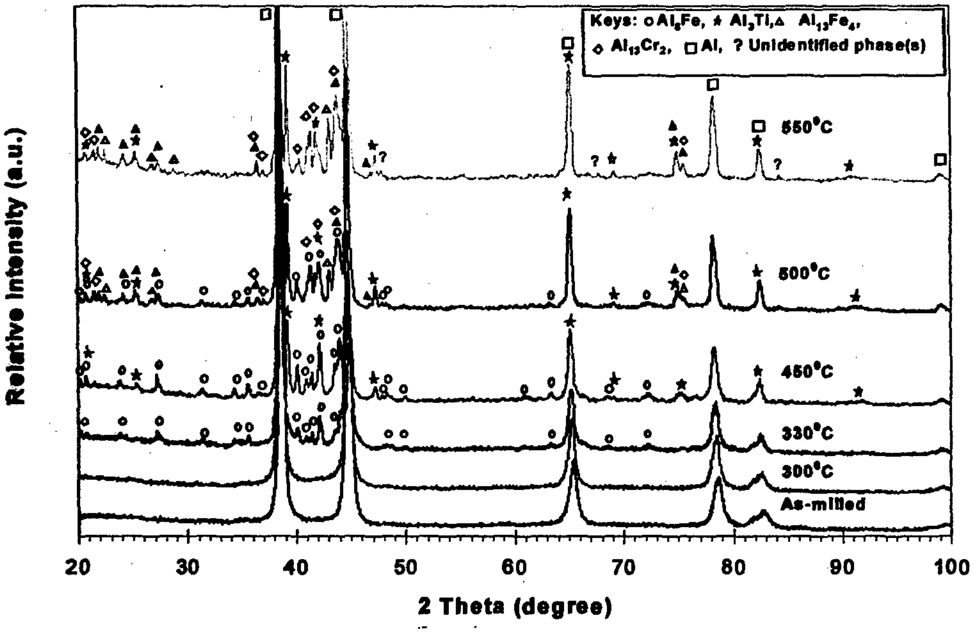


Fig. 4 XRD patterns of the mechanical alloyed Al powder as a function of annealing temperature with 1-hour holding time at the temperature indicated.

Shown in Fig. 5 is a typical TEM image of the extruded material, indicating that the grain size of the mechanical alloyed $Al_{93}Fe_3Ti_2Cr_2$ is still below 100 nm after extrusion. The compressive stress-strain curves of the extruded $Al_{93}Fe_3Ti_2Cr_2$ are shown in Fig. 6. It is quite clear that the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ has superior strength with excellent ductility at both ambient and elevated temperatures under compressive loading conditions.

Fig. 7 compares the compressive strength of the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy with the tensile strengths of two typical coarse-grained Al alloys, one being 7075 Al – one of the best Al alloys for ambient temperature applications, and the other being 2219 – one of the best Al alloys for high temperature applications. Included in Fig. 7 are also the tensile strengths of the future aluminum alloys that have the ratio of the ultimate strength to density equivalent to that of Ti-6Al-4V alloy. It is clear from Fig. 7 that the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy is much stronger than 7075 and 2219 alloys at both ambient and elevated temperatures. Furthermore, for lightweight, high strength structural applications at temperatures below 300^0C, the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy competes very favorably against the strength of Ti-6Al-4V.

Fig. 5 A TEM bright-field image of the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy after extrusion.

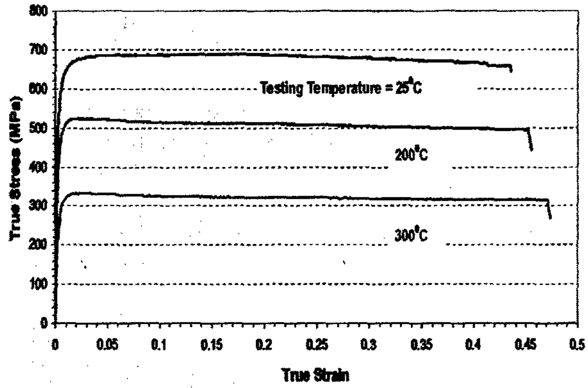


Fig. 6 True stress-strain curves of the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy obtained from compressive tests.

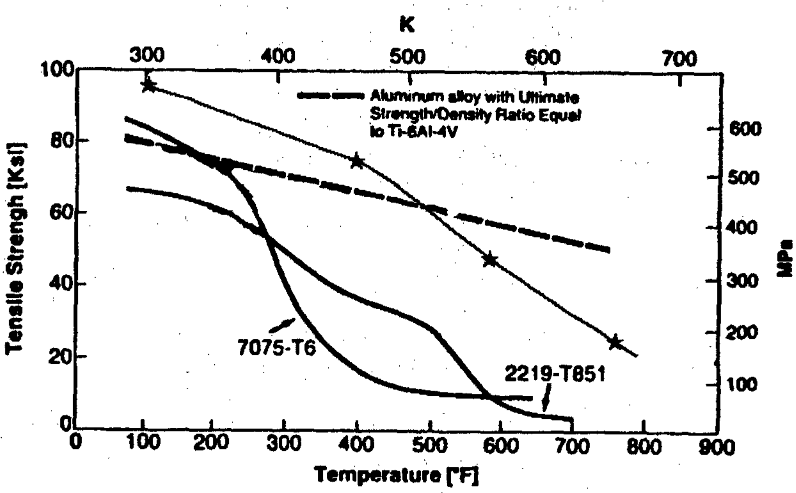


Fig. 7 Strength comparisons between the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy and commercial Al alloys. Compressive strengths are shown for the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy (★), whereas tensile strengths are presented for all the other alloys.

CONCLUDING REMARKS

The present set of experiments clearly indicates that nanocrystalline $Al_{93}Fe_3Ti_2Cr_2$ alloys can be prepared via mechanical alloying starting from elemental powders. The supersaturated Al-based solid solution formed from mechanical alloying is stable up to 300^{0}C above which precipitation occurs. Grain growth has also been observed when the MA-processed Al alloy is exposed to elevated temperatures. Nevertheless, nano-grains are retained after extrusion. Promising mechanical properties (i.e., superior compressive strength and ductility) at both ambient and elevated temperatures have been achieved. The superior high-temperature strength of the MA-processed $Al_{93}Fe_3Ti_2Cr_2$ alloy is likely due to low diffusivities of the alloying elements, the presence of nanoscale intermetallic precipitates, the formation of supersaturated solid solutions,

and the retention of ultrafine grains (fcc-Al < 100 nm). Tensile properties of this MA-processed alloy are currently under investigation and will be published in a forthcoming paper.

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REFERENCES

1) Y. He, S. J. Poon and G. J. Shiflet, Science, 241, 1640-1642 (1988).
2) A. Inoue, K. Ohtera, A.-P. Tsai and T. Masumoto, Jpn. J. Appl. Phys., 27 [3] L280-L282 (1988).
3) A. Inoue, K. Ohtera and T. Masumoto, Jpn. J. Appl. Phys., 27 [5] L736-L739 (1988).
4) G. J. Shiflet, Y. He and S. J. Poon, J. Appl. Phys., 64 [12] 6863-6865 (1988).
5) A. Inoue and H. Kimura, Mater. Sci. Eng., A286 [1] 1-10 (2000).
6) A. Inoue and T. Masumoto, Mater. Sci. Eng., A133, 6-9 (1991).
7) H. Chen, Y. He, G. Shiflet and S. J. Poon, Scripta Metall. Mater., 25, 1421-1424 (1991).
8) A. Inoue, Mater. Sci. Eng., A179-A180, 57-61 (1994).
9) Z. C. Zhong, X. Y. Jiang and A. L. Greer, Mater. Sci. Eng., A226-228, 531-535 (1997).
10) J. Q. Guo and K. Ohtera, Acta Mater., 46 [11] 3829-3838 (1998).
11) A. Inoue, Mater. Sci. Forum, 235-238, 873-880 (1997).
12) A. Inoue, H. M. Kimura, K. Sasamori and T. Masumoto, Mater. Trans., JIM, 36 [1] 6-15 (1995).
13) A. Inoue, H. Kimura, K. Sasamori and T. Masumoto, Mater. Trans., JIM, 37 [6] 1287-1292 (1996).
14) R. W. Hayes, R. Rodriguez and E. J. Lavernia, Acta Mater., 49, 4055-4068 (2001).
15) T. B. Massalski (Eds), in Binary Alloy Phase Diagrams, 2nd edition, ASM International, Materials Park, OH, 1990, pp. 147-149; pp. 138-140; pp. 225-227.
16) H. Mehrer (eds.), Diffusion in Solid Metals and Alloys, Landolt-Bornstein, New Series, Group III, Volume 26 (Springer-Verlag, NY, 1990).
17) C. Wagner, Z. Elektrochem., 65, 581-591 (1961).
18) D. L. Erich, Development of A Mechanically Alloyed Aluminum Alloy for 450-650^{0}F Service, AFML-TR-79-4210, (1980).
19) L. Shaw, M. Zawrah, J. Villegas, H. Luo and D. Miracle, Metall. Mater. Trans., in press.
20) L. Shaw, J. Villegas, H. Luo and D. Miracle, Acta Mater., in press.
21) L. Shaw, H. Luo, J. Villegas and D. Miracle, to appear in Microstructural Design of Advanced Materials, M. A. Myers, R. O. Ritchie and M. Sarikaya (eds.), Elsevier Science, Amsterdam, Netherlands.